

Title: Gas generator and method for the generation of low-temperature gas

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The invention relates to applied chemistry, more specifically to a composition for the generation of gases of low temperature and a process for the obtaining of gases of low temperature.

Gas generating processes based on the decomposition or burning of chemical propellants and other compositions are frequently being used for a number of purposes such as the inflation of airbags from, for instance, cars, rafts, life boats and vests, fast installed partitions (which are used in well drifts to cut off the well in case of fire), drives and generators for different types of pneumatic systems and operations mechanisms etc.

Some technical methods for obtaining relative cold gases, in particular nitrogen, are known. These methods are based on the decomposition or the burning of solid materials in special units. These materials are generally shaped in the form of monolithic or porous products and come in all types of shapes and sizes.

The hot gases generated from the decomposition of these materials are in general cooled with the aid of special chemical cooling agents or by specific designed features such as heat exchangers.

The high temperature burning gases are passed through the layer of the cooling agent or the heat exchanger and the temperature of the gases decreases as a result of the endothermal decomposition process of, or heat absorption by the cooling agent. Such processes are described for instance in US-1362349, GB-1371506, FR-136897 and the Russian inventors certificate 801540. The use of heat exchangers is described in GB-1500137 and GB-1487944.

The degree of cooling of the generated gas depends on the nature of the cooling agent, the mass of the cooling agent, which can sometimes exceeds

the mass of the gas-generating composition, and in case of the heat exchanger, the design features of exchanger.

One of the drawbacks of the prior art as cited above is the relatively complicated structure of these units. Another drawback is that the known gas generators do not allow or provide for the gases to be cooled below 150°C. Therefore the applicability of these gas generators is limited to systems that can withstand such high temperatures. These are disadvantages from cost-economic and application viewpoints.

Additionally, gases obtained by the use of the above described methods contain large and undesired amounts of components which may not only have a negative effect on the construction but also in case of airbags for cars, for the person (driver) who is supposed to be protected by the airbag.

Complicated design and complex products resulting in their increasing mass, size and complexity are negative features of these gas generating methods. This decreases reliability and efficiency of the complete system. Especially in the life saving airbags industry there is a continuous need for reliable, safe and economic methods for the generation of cold gases.

RF-patent 2108282 describes a method of generating cold gases, specifically nitrogen, but also hydrogen and oxygen, by using the endothermal decomposition of a product made of gas penetrable solid material. The gas penetrable solid material comprises a gas source and a heat absorbing mixture, whereby the gaseous reaction products are cooled by passing the hot gases through the porous body of the product in the moving direction of the reaction front. The hot gases heat the porous body to a temperature necessary to support the endothermic chemical reaction taking place. The heating of the porous body is necessary to enable the main reaction. The decomposition of the cooling agent is also an endothermic chemical reaction. The patent claims to obtain nitrogen gas from a solid propellant system with a purity better than 97% and a temperature below 150°C.

In the gas generator using this method (as well as in most other gas

generators) azides, hydrides and chlorates are used as the gas source, which compounds are in general used in the form of alkali and earth-alkali compounds. On decomposition of these compound usually a highly reactive metallic slag remains behind in the gas generator.

5 As an example, for a nitrogen producing gas generator composition NaN_3 may be used. The decomposition reaction of NaN_3 results in Na and N_2 . Likewise in other decomposition reactions of sodium compounds, also sodium is formed. The formed gas is blown off and the slag remains. This slag comprises of the remains of cementing agent and , the cooling agent and the
10 metallic sodium. Under these conditions of gas generation the highly chemical reactive sodium is thus generated. This highly reactive material will accumulate in the condensed burning decomposition products and thus provides a potential hazard for persons involved. When moisture is present this can result in vigorous and dangerous reactions taking place in
15 combination with the generation of the highly flammable and explosive hydrogen. The decomposition of which might be followed by explosions, other undesirable effects or even personal injuries, if persons are involved.

 Methods for the neutralisation of sodium are itself known in the art and for instance described in "Sodium production, its properties and use",
20 State Publishing House, Moscow, 1961 pp 142. One of the methods described for the removal of metallic sodium is destruction with water. To be able to apply this method in order to neutralise the used gas generator, the generator after use has to be hermetically sealed and transported to a suitable installation to adequately neutralise the reactive remains of the generator.
25 This is dangerous, cost-ineffective, complex and thus undesirable.

 In the case of sodium-compounds as the gas source, elemental sodium (Na) is formed upon decomposition of sodiumazide. Sodium is a highly reactive and aggressive chemical. As a result of this reactivity, sodium can react with a wide class of substances to a number of sufficiently stable
30 compounds. One of these compounds is sulphur. Sodium reacts with sulphur to

form sodium sulphide (Na_2S).

The neutralisation of sodium by reaction with sulphur or sulphur compounds in gas generating compositions is known for instance from US 3775199, US 5536340, EP 394103 and US 3741585. The sulphur is vaporised during the decomposition of the gas-generating composition and reacts with the formed sodium slag to the neutral sodiumsulphide.

In the gas generators of the prior art as described hereinabove, the sulphur is vaporised together with the gas generation. It is difficult to vaporise the sulphur at the same rate at which the sodium slag is formed and the rate at which it reacts with the sodium slag. As a result vaporised sulphur will exit the gas generator and/or not all metallic sodium is neutralised. This is a drawback of the use of mixtures of sulphur and gas-generating compositions as described in the prior art.

It is therefore a goal of the present invention to develop a product which will result in the effective generation of nitrogen gas of low temperature without the adverse effects as described above and without major concessions towards output and performance parameters of the gas generator.

It is another goal of the invention to provide for a process for the generation of nitrogen gas of low temperature and to provide for a gas generator which generates nitrogen gas of low temperature.

Inventors have now found a gas-generating configuration that can overcome the above-mentioned deficiencies of the prior art and results in the generation of low temperature gas with effective and sufficient neutralisation of the reactive slag.

The invention accordingly comprises a gas generator comprising at least one first body, comprising means for the generation of gas, and at least one second body, comprising means for the generation of a neutralisation agent, wherein means are present for contacting the said neutralisation agent with the said first body, to neutralise reaction products from the generation of gas in the said first body, and wherein means are present for operating the

generation of a neutralisation agent in the second body at a temporal and/or spatial interval from the generation of gas in the first body.

The principle of the invention encompasses the separation of gas generation material and neutralising material, thereby making it possible to improve the effectivity and reliability of the gas generation and neutralisation. According to an embodiment of the invention, two gas generating materials are present in one housing, spatially separated from each other. A first gas generator with the primary task of generating gas, preferably of low temperature, and a second gas generator with the primary task of generating neutralising compounds for the slag obtained from the first gas generator.

The first gas generator comprises a composition from which nitrogen, hydrogen and/or oxygen gas, preferably of low temperature can be obtained by the decomposition of a gas generating composition in the form of a gas penetrable solid material wherein the generated gaseous products are passed through the porous body in the direction of the moving decomposition front.

The second gas generator (the neutraliser) is another composition generating a neutralising gas, preferably comprising a gas generating composition together with an effective neutraliser compound, for instance sulphur, iron oxide, metal sulphide, metal oxides (from Fe, Cu, Mg, Ti, Sn, B etc.), SiO_2 and the like. With the neutraliser composition a neutralising gas is generated separately from the gas generated in the first generator. The neutralising gas is generated at a time and/or space interval with the first gas generator. It is an important aspect of the invention, that the neutralising agent does not come into contact with the decomposing solid porous material, during or prior to the decomposition thereof. The invention is based on the principle, that only after the material has been decomposed, the neutralising material is passed through the decomposed porous solid material, thereby neutralising the (usually hazardous) decomposition products (slag). The neutralising gas is generated at a rate and a manner that the effective

neutralisation of slag is accomplished and the vaporous neutralising agent is not emitted. The neutralising agent, such as vaporous sulphur, reacts with the reaction products (slag) from the first gas generator such that the these products are effectively neutralised.

5 In an embodiment the invention thus relates to a first gas generator comprising a gas penetrable solid material comprising a nitrogen source, preferably an azide, more preferably sodiumazide, cementing agent and optionally a heat absorbing mixture, wherein the solid material has a porosity of 35-60 % and a second gas generator containing a neutraliser composition
10 which contains sulphur and an additional nitrogen source.

The gases to be generated can be selected from the group of nitrogen, oxygen and hydrogen, or combinations thereof. Generally azides, hydrides and chlorates are used for that, preferably in the alkali metal form.

15 In a further embodiment of the invention the gas to be produced is nitrogen, the nitrogen sources in both the first and the second gas generator are selected from the group of alkalimetal azides or an earth-alkalimetal azides, preferably potassium azide or sodium azide, more preferably sodium azide.

20 The first and second gas generator do not have to be physically separated from each other. In embodiments of the invention they can be placed in any position relative to each other, as long as the vaporised neutraliser of the second generator can come into contact with the slag from the first generator.

25 In the invention, the neutralisation takes place behind the reaction front of the decomposition reaction of the first gas generator. The spatial interval between the said reaction front of the first gas generator and production of the neutralising agent in the second gas generator is such that the reaction products of high temperature from the first gas generator stay behind, while the nitrogen gas is blown off. The neutralisation front lags
30 behind the decomposition front and neutralises the said reaction products

remaining behind.

In another embodiment of the invention the rate at which the gas generating composition decomposes is different from the decomposition rate of the neutraliser charge. Thus, the decomposition of the gas generating composition and the neutraliser are started simultaneously. Metallic slag is formed, followed by the generation of vaporous neutraliser in the second generator, which neutralises the slag.

In another embodiment of the invention the moment at which the neutraliser is activated lies later than the moment of activation of the gas generator.

The activation, or ignition, of the two bodies can be done by any suitable means known in the art.

A typical embodiment of the invention is as follows.

A body consists essentially of two parts: the gas generator and the neutraliser. The gas generator will contain a porous solid material, containing a gas generating component such as sodiumazide, together with cementing agents (such as phenolic resins) and optionally cooling agents or other heat absorbing mixtures. The other part of the body is the neutraliser mass. The neutraliser contains the neutraliser (sulphur, iron, metal sulphides, metal oxide) and a gas generating component. The gas-generating component may be identical to the gas generating component in the first part, for instance sodiumazide. When the gas generator is activated, gas is generated and blown off, leaving behind highly reactive metallic sodium slag. The neutraliser is activated and the neutralising reagent is vaporised; in the case of solid neutralising agents it may be brought in aerosol form. The neutraliser will react with the slag, resulting in non-hazardous or less hazardous materials, in the case of neutralising sodium with sulphur, resulting in the neutral sodiumsulphide.

The amount of neutraliser is such that it is sufficient to effectively neutralise the slag formed in both the neutraliser and the gas generator and

that only minimal or almost no vaporous neutraliser is blown off.

In the present invention, in order to facilitate the interaction between the sodium and the neutraliser compound (e.g. sulphur) it is preferred that the neutralisation product is in a form in which the reaction with the sodium slag is enhanced. To this extent the neutraliser can be mixed with the gas-generating compound in the form of powder, granules, etc.

In a gas generator according a preferred embodiment of the invention, said gas generator being based upon the use of sodium azide and sulphur, the combined amounts of the nitrogen sources in the first and second body comprises 50-80 wt.% drawn on the total weight of the gas generator and the amount of neutralisation agent in the second body 47-90 wt.% of neutralisation agent, drawn on the weight of the second body. The respective weight of the gas generator is measured in the absence of housing, external cooling aids, etc.

The second body (gas generator) comprises between 17 and 35 wt.% of the gas generator according to the invention, drawn on the total weight of the gas generator. The second body (gas generator) contains 10 to 53 wt.% of the nitrogen source and 47 to 90 wt.% of neutralising agent. In a preferred embodiment the second body (gas generator) contains 15 to 25 wt.%, more preferable 17 to 23 wt.% of nitrogen source and 75 to 85 wt.%, more preferable 77 to 83 wt.% of sulphur.

In a preferred embodiment the sulphur is in a particulate form, preferably in the form of small particles, more preferably in the form of sulphur powder.

The relative amounts of sodium azide and sulphur are contained between the lower limit of sulphur which that is the amount of sulphur necessary for the neutralisation of the elemental sodium formed. The upper limit of sulphur is determined by the amount at which almost no vaporised sulphur will be blown off or the amount that is considered acceptable with respect to output gas purity.

The rate at which the gas was generated was determined in order to provide for an optimal formulation together with the optional heat absorbing product and the neutraliser product. The ratio of the different components (nitrogen source, heat absorbing material and sulphur) was chosen such that the required maximum discharge of vaporised sulphur and the stable burning of the material was obtained. It was found that a stable ignition and burning of the material was not possible if the concentration of the sulphur in the material was more than 90 wt.% of the combined weight of additional nitrogen source and sulphur (neutraliser mass). If the concentration of sulphur was below 47 wt.% of said combined weight, the discharge of vaporised sulphur decreased below the desired level and the total (neutraliser mass)/(nitrogen source) ratio had to be increased in order to obtain the bonding of the elemental sodium in sufficiently high levels. The preferred mass ratio of the nitrogen source and the neutraliser is determined by the total neutralisation of sodium to sodium sulphide in the slag.

In a preferred embodiment of the invention the nitrogen source and the neutraliser, preferably sulphur, are homogeneously mixed as part of the second body.

In another preferred embodiment of the invention, the neutraliser product comprises sulphur and additional nitrogen source in an amount of 10-53 wt.% of the additional nitrogen source and 47-90 wt.% of sulphur, based on the combined weight thereof.

In this embodiment of the invention, the combined amount of the nitrogen source and sulphur, based on the total weight of the product is from 17 to 35 wt.%.

In case the combined amount of the additional nitrogen source and the sulphur is less than 17 wt.%, the total neutralisation of sodium is insufficient because of lack of sulphur. In case the amount is above 35 wt.%, the vaporised sulphur will be blown off with the generated gas and thus the purity of the generated nitrogen gas decreases.

It is to be noted that in some cases the generated gas may contain some entrained contaminants. If these are un-desirable in the intended use of the generator, it may be advantageous to include additional downstream filter means. This may be any kind of filter, such as sand, chemical filters, metal
5 wire filters and the like. In some instances it may also be advantageous to include some additional neutralising agent in the filter, thereby providing an additional safeguard against contaminants being blown out with the gas.

In the case of generating a cold gas by passing the generated gas through the porous solid material, as described above, the situation may occur
10 that when the material is almost completely decomposed, the cooling capacity of the remainder of the porous material is too small to maintain the temperature of the gas at a constant level. If in a specific application this is not acceptable, it can be advantageous to include downstream cooling means in the gas generator. It is possible to combine these cooling means with filter
15 means discussed above, especially as both the cooling means and the filter means can easily be constructed from the same materials (sand, steel wire, steel wool, metal mesh and the like).

The invention also relates to a process for the generation of gases, preferably nitrogen, comprising the steps of:

- 20 - decomposition of a gas-penetrable porous solid material in a first body, whereby gas and other reaction products are generated at a decomposition front;
- generating a neutralisation agent in a second body;
- neutralising the said other reaction products in the first body by
25 reaction with the neutralisation agent;
- maintaining a temporal and/or spatial interval between the decomposition front of the first body and a neutralisation front obtained by passing the neutralisation agent from the second body into the first body.

Upon ignition of the nitrogen source containing gas generating
30 material and the neutralisation material; the materials start decomposing.

The gaseous decomposition products of the nitrogen source pass through the ramified porous body in the moving direction of the reaction front and are cooled by transferring heat to the porous body. At the burning of the neutraliser, vaporised sulphur is generated and passed through the slag of the nitrogen source. In an embodiment of the invention a spatial and temporal interval between the reaction front of the nitrogen source and the reaction front of the neutraliser is provided. The reaction between the vaporised sulphur and the metallic sodium is exothermic. However, as there is a spatial and/or temporal interval between the gas generation and the neutralisation, this will not influence the temperature of the generated gas. This interval can be accomplished by a lower reaction rate of the neutraliser when compared to the reaction rate of the nitrogen source or by a suitable time delay. By this interval the vaporised sulphur is mainly generated after the sodium is formed, thus allowing for more optimal reaction conditions for both the generation of gas and the neutralisation of sodium.

The interval can also be controlled by design related features such as the adjustment of the flow rates by a different form of the burning surface or by the non-simultaneous ignition of the nitrogen source and the neutraliser. The invention accordingly comprises a generator for low temperature gas.

In a preferred embodiment the generated gases are cooled by passing the gases through a porous body in the moving direction of the reaction front.

In an preferred embodiment heat is absorbed which is formed in the exothermic reaction by a heat absorbing material included in the porous body.

In a preferred embodiment of the invention the amount of heat generated in relation to the amount absorbed heat is such that the generated gas is cooled to a temperature below 150°C, preferably below 100°C.

The invention is now elucidated on the basis of the attached figure. In the figure a gas generator is shown, having a housing 1, provided with an opening 2, for generated gas. In the housing 1, two gas generating bodies 3,4

are present. A first solid porous body 3, providing the major amount of gas, and a body 4, providing a neutralising gas. Further a body 5 of cooling and/or filter material is present, for example a sand filter, optionally containing a dispersed additional neutralising agent.

5 Once the body 3 has been ignited by igniting means (not shown), the decomposition starts, resulting in the production of a gas, which flows mainly in the direction of the arrows B, i.e. through the body 3, thereby heating the porous material, at the same time as being cooled to a relatively low temperature. Finally the cooled gases leave the housing 1, through opening 2
10 in the direction of arrows C.

The decomposition of the porous solid material proceeds with time and the decomposition front moves in the direction of arrow A.

From body 4, a neutralising gas is produced, after ignition of the body (by ignition means, not shown). The gas flows in the direction of arrows D
15 and creates a neutralisation front (not shown) in body 3, which front stays behind the decomposition front, but moves in the same direction (arrow A).